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THE VISCOSITY OF TATB TYPES A AND B
SUSPENSIONS IN MOLTEN TNT: GENERAL
CHARACTERISTICS

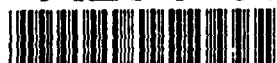
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H. H. BILLON AND M.A. PARRY

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The Viscosity of TATB Types A and B Suspensions in Molten TNT: General Characteristics

1. Introduction

In recent years there has been increasing emphasis placed on the development of explosives that are insensitive to accidental hazardous stimuli [1]. One of the most important of these explosives has been 1,3,5-triamino-2,4,6-trinitrobenzene (TATB), which has the added advantage of high thermal stability. Presently TATB is in military use in both the US and UK, and is also an explosive component in some polymer bonded explosive (PBX) compositions (e.g. PBX-9502, (TATB / Kel-F chlorotrifluoroethylene polymer 95:5)) used to prepare pressed charges.

Because Australia still has a substantial production capability in melt-cast explosives technology, a desirable way to lower the vulnerability of locally manufactured ordnance would be to develop insensitive high explosive formulations that can be processed in existing equipment (perhaps with modifications). Research is being conducted at MRL on cast-cured PBXs as one means of achieving this [2]. The other alternative is to replace the RDX in currently used melt-cast fillings such as Composition B (RDX/TNT/beeswax 60:40:1) or H-6 (RDX/TNT/A1/wax) by a less sensitive explosive filler such as TATB.

In a 1969 study in the US on insensitive high explosives and propellants (IHEP) [3], two melt-cast formulations containing TATB were considered:

- (1) TATB/TNT 50:50
- (2) TATB/TNT/A1 40:40:20

However, virtually no information has been reported on these explosive compositions [4].

As part of our studies of the rheology of molten TNT suspensions [5] and insensitive high explosives we investigated the behaviour of TATB in molten TNT. A characterisation of the rheology of TATB/TNT suspensions will assist in the development of a composition with maximal solids loading and acceptable rheology. This will enhance processability and minimize hazard

response. Knowledge of the rheology will also facilitate the design/operation of plant equipment.

This paper is a summary of the general rheological characteristics of TATB in molten TNT as well as a discussion of problems encountered and recommendations for further research.

2. Factors Influencing Suspension Rheology

Evaluating the rheology of suspensions usually includes acquiring a knowledge of the following:

- (1) the volume fraction of the disperse phase,
- (2) the shear rate (stress) applied to the suspension,
- (3) the particle size distribution of the disperse phase,
- (4) the particle shape of the disperse phase, and
- (5) the maximum packing fraction of the disperse phase

It should be noted that (5) is determined by (3) and (4).

Viscoelasticity can be important in some cases but is apparently unimportant in TATB/TNT mixes as there is no significant recoil in pouring. The rheological behaviour of suspensions is often complicated by time-dependent behaviour (thixotropy). If the material is thixotropic the flow behaviour must be modelled thoroughly to enable prediction of even simple flows. Some TATB/TNT suspensions are thixotropic (see later) but thixotropic modelling was not attempted in this study.

3. Experimental

3.1 Description of Materials

Type A and type B TATB were used. Both types were generously donated by Royal Armaments Research and Development Establishment (RARDE), Sevenoaks, Kent, UK (now DRA Military Division). The TNT was commercial grade from Albion Explosives Factory. The median diameters of types A and B were 40 μm and 20 μm respectively. RDX Grade B with a median diameter of 140 μm was also used in some experiments.

3.2 Description of Viscometric and Mix Apparatus

A Haake RV2 rotational viscometer was used. A Haake PG 142 programmer drove the viscometer under a given speed regime. The sensor system was either a smooth surfaced MVII or a profiled SVIIP system. The bob radii of the

MVII and SVIIP systems (Fig. 1) are 18.4 mm and 10.1 mm respectively and both have a cup to bob radius ratio of 1.14. The samples were enclosed by a water jacket and held at $95.00 \pm 0.01^\circ\text{C}$ by a Haake N3 bath.

A jacketed melt and mix kettle of 95 mm internal diameter and 0.5 L capacity was used for sample preparation. Incorporation was by a 12-vaned impeller of 63 mm diameter driven by a Janke and Kunkel stirrer. The kettle was held at 95.00°C by the N3 bath.

3.3 Sample Preparation for Viscosity Determination

The solids (TATB or RDX) were incrementally added to molten TNT in the kettle at 95°C and incorporated by the impeller turning at 200 r/min. After the solids seemed well dispersed in the TNT a further 30 min of stirring was performed at 200 r/min to ensure that no aggregates of solids and resolidified TNT remained. The sample was then transferred to the viscometer. Incorporation of the TATB and RDX into molten TNT required 60 min and 40 min respectively.

3.4 Experimental Procedure for Viscosity Determination

3.4.1 Rotational Viscometry

The bob and cup of the appropriate sensor system were kept in an oven at a temperature of 100 to 105°C . The cup was then filled with the mix from the kettle. The bob was attached to the measuring head and the cup inserted into the temperature-controlled water jacket of the viscometer.

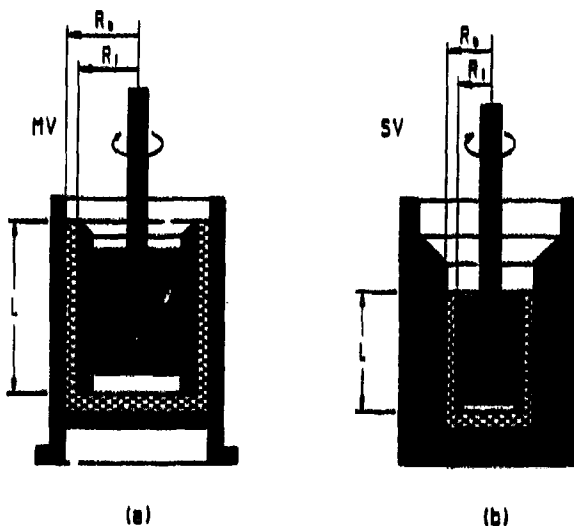


Figure 1: Depiction of (a) Haake MVII sensor system, (b) Haake SVIIP sensor system.

3.4.2 Efflux Viscometry

This consisted of recording the time taken for 25 mL of sample to flow through an orifice of radius 3.2 mm and depth 15 mm [6].

3.5 Settling Experiments

A TATB blend was mixed with powdered TNT. This was placed in a 10 mL measuring cylinder in a jacketed kettle filled with silicone fluid at 95°C. The kettle was agitated on a vibrating table until the TATB bulk volume was constant. The maximum packing fraction was calculated from this volume.

4. Results and Discussion

4.1 The Effects of Varying Concentration

TATB/TNT suspensions at low concentrations exhibit Newtonian behaviour. This can be seen in Figure 2 (a) and (b) for a suspension of 20 wt % TATB type B in TNT. The flow curve in 2 (b) was obtained 1.5 h after that in 2 (a). The observed 'bends' in the flow curves can be correlated with transitions from laminar behaviour at high rotation speeds. They are similar to those found in molten TNT [7]. The product (density) \times ("bend" r/min)/(viscosity) is proportional to the Reynolds number and is 23.3 for curve 2 (a) and 24.1 for curve 2 (b) (assuming density = 1.53 g/mL). Thus the Reynolds number is approximately constant indicating that these "bends" are the result of departures from laminar flow.

The behaviour of suspensions at high concentrations is very different. Figure 3 (a) depicts a 51.5 wt % TATB type B suspension and Figure 3 (b) depicts a 51.5 wt % TATB type A suspension in TNT. Both thixotropy and the presence of a yield point are evident as is the lack of flow curve linearity.

4.2 Aging of TATB/TNT in the Viscometer

TATB/TNT suspensions do not thicken noticeably when held in the viscometer for long time intervals. Figure 4 depicts transient stress curves taken 7 h apart. The curves were obtained by shearing the sample to constant stress. There is no significant change in apparent viscosity on aging because the low solubility of TATB in molten TNT at 95°C prevents significant dynamic recrystallisation.

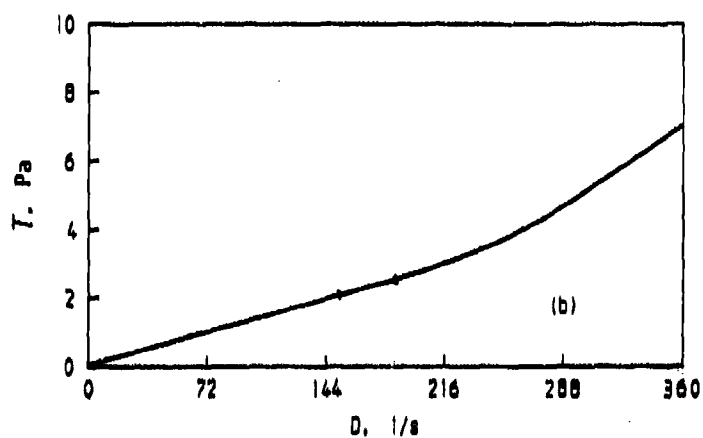
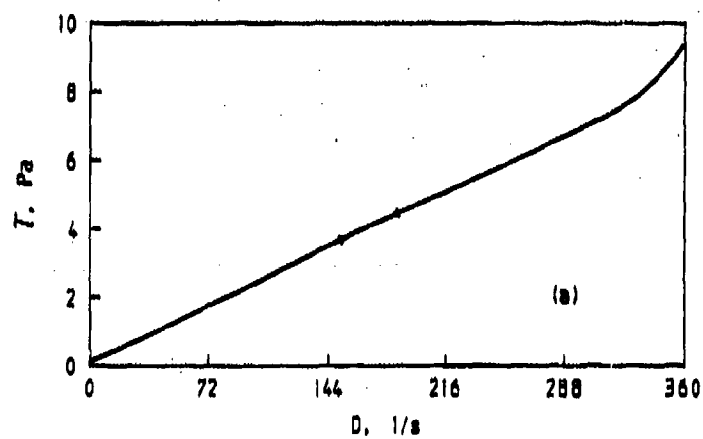


Figure 2: (a) First flow curve, (b) second flow curve for a TATB/TNT 20/80 suspension at 95°C. Sample sheared from 0 to 400 r/min in 4 min, held at 400 r/min for 0.1 min then back to 0 r/min in 4 min. MVII sensor system.

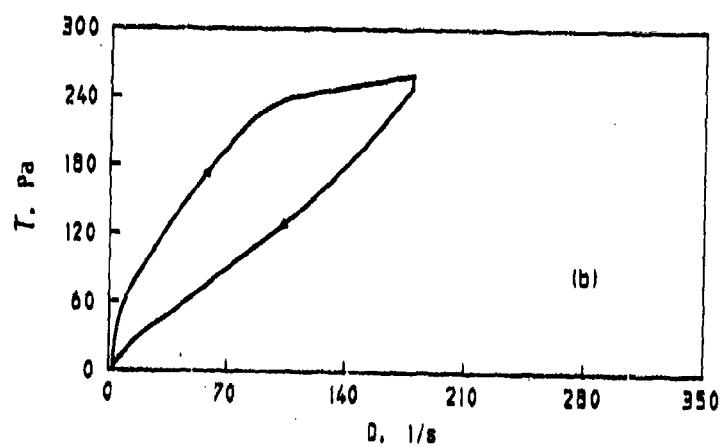
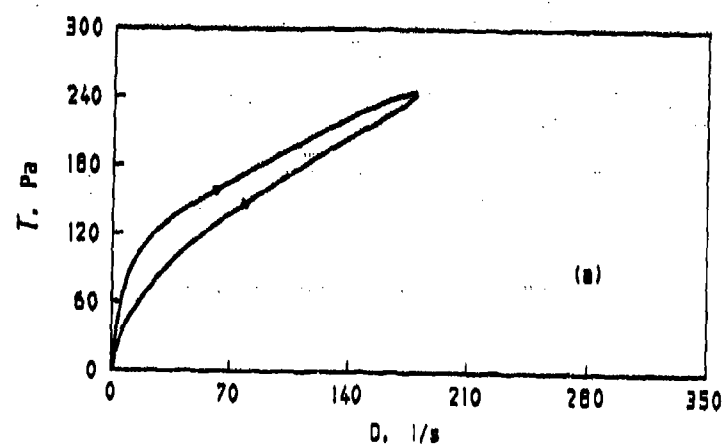


Figure 3: Flow curves for (a) TATB type B, (b) TATB type A suspensions at 95°C. TATB/TNT 51.5/48.5 suspensions sheared from 0 to 231 r/min in 2 min, held at 231 r/min for 0.1 min then back to 0 r/min in 2 min. - SVIIP sensor system.

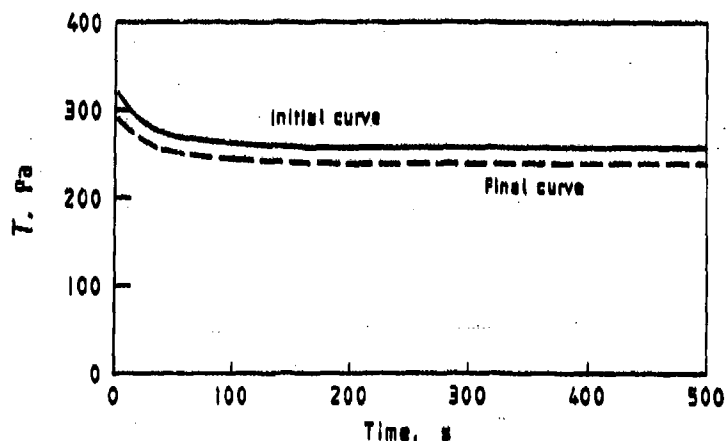


Figure 4: Transient stress curves obtained 7 h apart at 95°C. TATB/TNT 70/30 sample sheared to equilibrium at 128 r/min. TATB type A. SVIIP sensor system.

4.3 The Effects of Blending on Flow Behaviour

To determine the effects of blending the two types of TATB experiments were conducted on weight ratios of types A to B of 100:0, 75:25, 50:50, 25:75, and 0:100. The wt. ratio of TATB to TNT was held constant at 51.5:48.5. As a reference system for comparing rheological properties 60:40 RDX/TNT was also investigated; it is almost identical to Composition B which is used for filling munitions.

The flow curves obtained immediately after incorporating and stirring the TATB into the molten TNT are the most important. This is because they afford a more accurate assessment of the flow properties than subsequent flow curves which may be affected by sedimentation, and also, in the case of RDX/TNT, by thickening due to recrystallisation [5]. Figures 5 and 6 depict apparent shear rate flow curves for the mixes mentioned above. These flow curves were obtained immediately after transferring the sample from the kettle to the viscometer. After baseline correction of the raw data a number of features were observed:

1. After stirring, the mixes containing only type A or type B TATB and the 75/25 and 25/75 A/B blends possessed yield points, while the 50/50 A/B and the RDX/TNT mixes did not, under these test conditions.
2. The yield points on the initial curve and return curves do not coincide for mixes with A:B ratios of 100:0, 75:25 and 25:75.
3. Blending the two types of TATB results in a viscosity decrease at constant solids level. This decrease in viscosity with blending was also observed in earlier work [8] (Table 1).

4. TATB/TNT suspensions which possess more apparent thixotropy (i.e. larger area of hysteresis loop) contain the greatest amounts of type A TATB, i.e. the 100:0 A/B and 75:25 mixes.

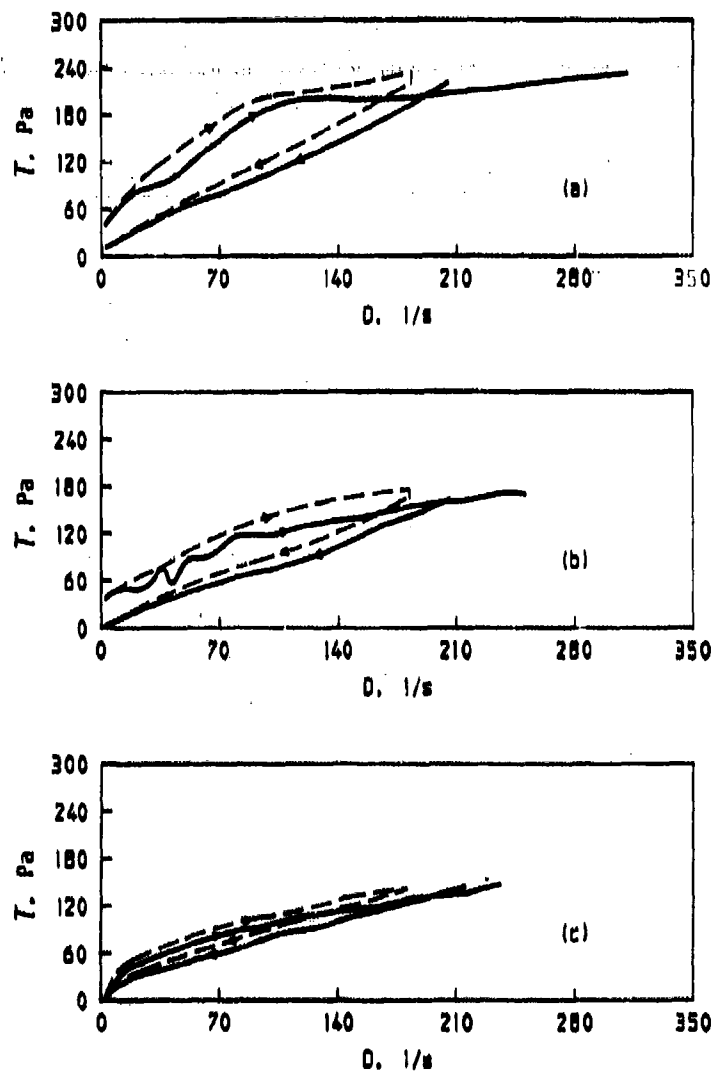


Figure 5: Flow curves for TATB/TNT 51.5/48.5 suspensions at 95°C. (a) TATB type A, (b) TATB A:B = 75:25, (c) TATB A:B = 50:50. Samples sheared from 0 to 231 r/min in 2 min, held at 231 r/min for 0.1 min then back to 0 r/min in 2 min. True flow curves: solid lines. Apparent flow curves: broken lines. SVIIP sensor system.

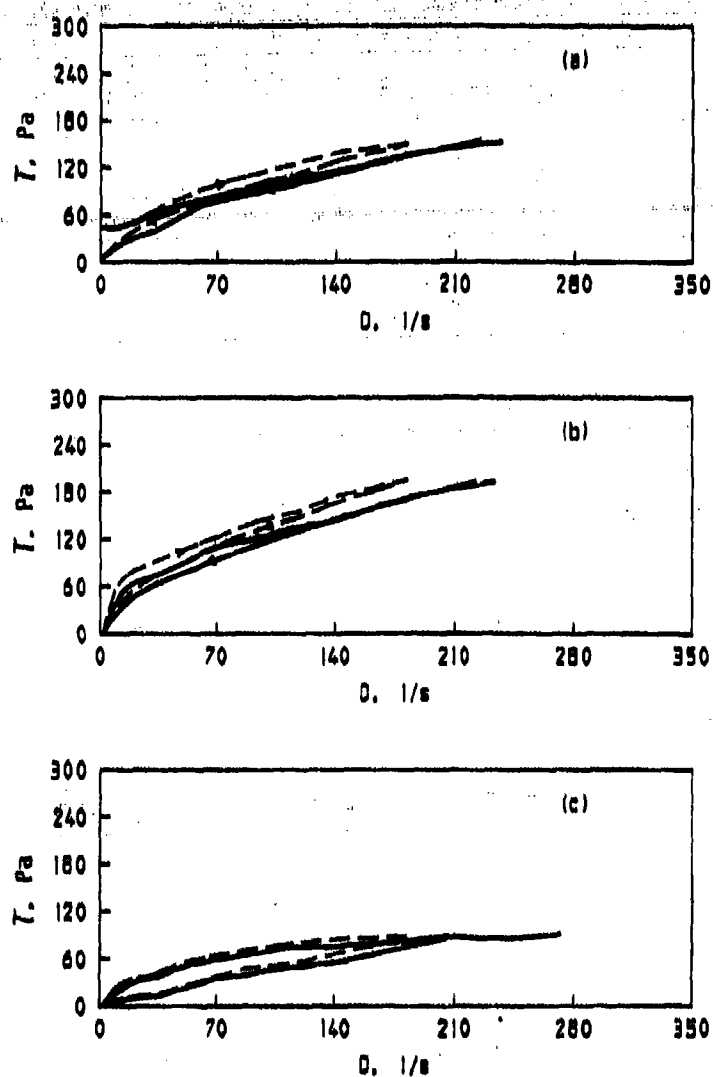


Figure 6: Flow curves for (a) TATB/TNT 51.5/48.5, TATB A:B = 25:75, (b) TATB/TNT 51.5/48.5, TATB type B, (c) RDX/TNT 60/40, RDX grade B suspensions at 95°C. Samples sheared from 0 to 231 r/min in 2 min, held at 231 r/min for 0.1 min then back to 0 r/min in 2 min. True flow curves: solid lines. Apparent flow curves: broken lines. SVIIP sensor system.

Table 1: Efflux Viscosity Results for TATB/TNT at 85°C

Run No.	TATB/TNT Composition	Av. Efflux Time (s) (Std. Dev.)	Remarks
98	10(A)/90	1	Sedimentation
99	20(A)/80	1.2	Some sedimentation
100	30(B)/70	1.7	Some sedimentation (Stirred at 200 r/min)
101	30(A)/70	1.8	Stirred at 300 r/min
102	40(A)/60	9.4	ET decrease with stirring
103	40(A)/60	9.1	ET decrease with stirring
104	50(A)/50	47.5	TATB added over 45 min at 300 r/min. Readings: 79.5 s, 39.5 s, 23.5 s
105	50(A)/50	40.5	Stirring increased to 600 r/min for 15 min before 1st reading and during readings. Readings: 64 s, 35 s, 22.5 s
106	40(B)/60	3.2	
107	40(B)/60	3.7	Pellet analysed 36.5 wt % TATB (suggesting sedimentation)
108	50(B)/50	50.6 (3.6)	Pellet 48.5 wt % TATB (stirring at 300 r/min)
109	50(B)/50	50.5 (5.1)	
110	25(A):25(B)/50	20.1 (3.4)	ET decrease 25.5 - 16.8 s with stirring 3 h 10 min at 300 r/min. (Pellet wt % TATB: 49.3, 49.0, 49.0, 49.0, 49.1)
111	25(A):25(B)/50	18.1 (4.0)	ET decrease 24.5 - 13.5 s with stirring 4 h 40 min at 300 r/min. (Pellet wt % TATB: 49.0, 48.7, 48.9, 48.8, 49.2)

Procedure: TNT was melted at 85°C, TATB was then added over about 0.5 hr while stirring at 200 r/min and was then stirred at 300 r/min (stirring speed was varied). A sample was then taken and the efflux time (ET) measured after about 25 - 30 min and then over about 6 hr (varied).

4.4 The Effects of Thixotropy on Shear History and Viscosity

To determine true shear rate from the data in section 4.3, plots of $\log \omega$ (angular velocity) versus $\log \tau$ (shear stress) were made (Figs 7 and 8). Lines were fitted to the straight sections of these plots. The fit regression coefficients

always exceeded 0.94 and were usually greater than 0.99. The true shear rate (in the absence of yield stress) could then be determined by two methods. The first employed the gradients of the $\log \omega$ versus $\log \tau$ plots as the flow behaviour index n in a power law approximation which uses the following equation for the shear rate of a system with radius ratio ϵ :

$$D = 2(\omega/n) \epsilon^{2/n} / (\epsilon^{2/n} - 1) \quad (1)$$

The second used the Krieger and Elrod technique [9]. The derivatives were determined from the fits to the $\log \omega$ versus $\log \tau$ plots. The discrepancy between the two methods was usually less than 0.1%. Only power law shear rates were used because of ease of determination.

The presence of a yield stress τ_y complicated shear rate determination for the case when:

$$\tau / \tau_y \leq \epsilon^2 \quad (2)$$

In this case the true shear rate at the bob is given by:

$$D = 2\omega/N \quad (3)$$

where N = the slope of the plot of $\log \omega$ versus $\log \tau$ [10].

The experiments involved programming the rotation speed from 0 r/min to 231 r/min in 2 min, holding at 231 r/min for 0.1 min and then programming down again to 0 r/min in 2 min. Thus all the test conditions were nominally the same. But on plotting the true shear rate as a function of time it was found that this was not really the case. Figures 9 and 10 reveal that the true shear history of a mix can bear little resemblance to the apparent shear history (calculated from the manufacturer-supplied constant) which is linear.

It is notable that the shear rate is not constant during the 0.1 min when the rotation speed is held constant at 231 r/min, but drops as listed in Table 2. This observation illustrates the common error of confusing constant rotation speed with constant shear rate for thixotropic substances. Both viscosity and shear rate can change while stirring at constant speed [10]. Figures 11 and 12 indicate a general decrease in viscosity for the return curve compared with the initial curve. However the viscosity increases at 231 r/min (Table 3).

Hysteresis loops for the true flow curves in Figures 5 and 6 are open and cross over. This phenomenon, absent in the apparent flow curves, stems from shear rate variation at constant rotation speed.

The shear rate analysis is incomplete because the Krieger and Elrod method [9], used for determining the true shear rate, was developed for time-independent samples. However the analysis indicates the stress and the viscosity changes with shear rate and characterises the differences between apparent and true shear rates.

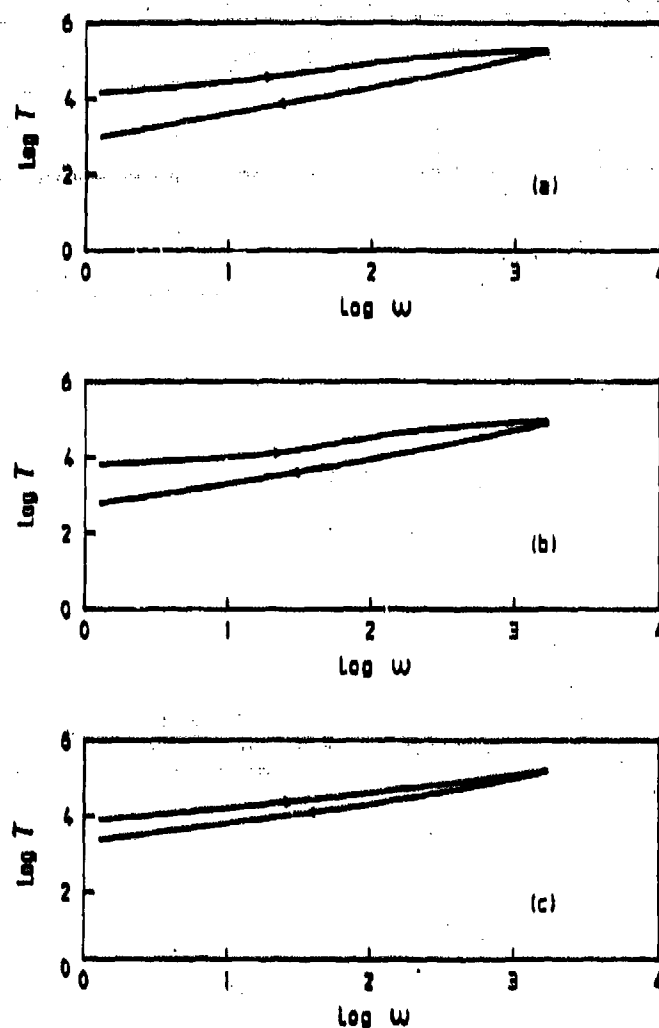


Figure 7: Log - log plots of shear stress vs angular velocity for TATB/TNT 51.5/48.5 suspensions at 95°C. (a) TATB type A, (b) TATB A:B = 75:25, (c) TATB A:B = 50:50.

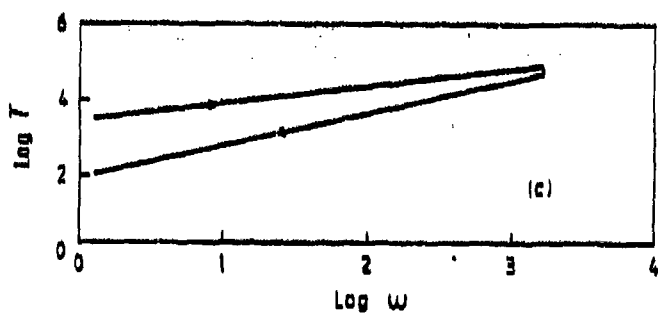
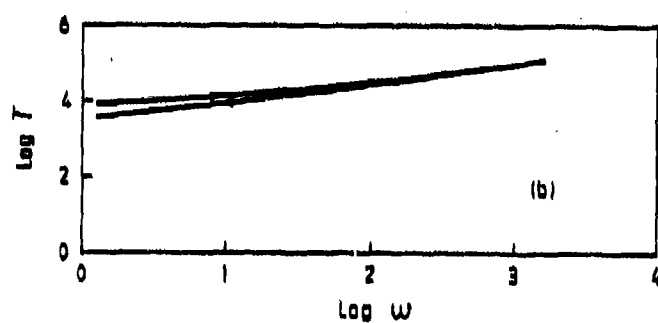
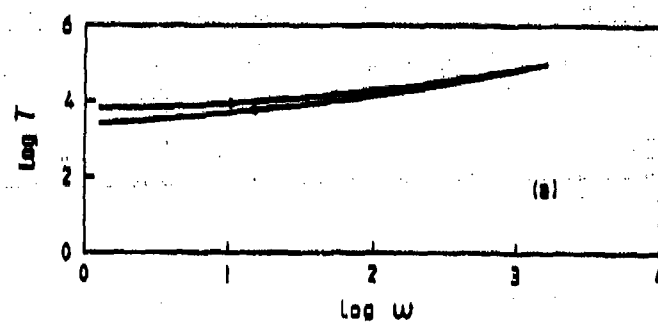


Figure 8: Log - log plots for (a) TATB/TNT 51.5/48.5, TATB A:B=25:75, (b) TATB/TNT 51.5/48.5, TATB type B, (c) RDX/TNT 60/40, RDX grade B suspensions at 95°C.

Table 2: Drop in Shear Rate at Constant Rotation Speed of 231 r/min.

TATB Sample (A:B Ratio)	Shear Rate at Start of 0.1 min (1/s)	Shear Rate at End of 0.1 min (1/s)
100:0	307	205
75:25	250	206
50:50	236	213
25:75	234	223
0:100	231	222

Table 3: Increase in Viscosity at 231 r/min

TATB Sample (A:B Ratio)	Initial Viscosity (Pa.s)	Final Viscosity (Pa.s)
100:0	0.78	1.13
75:25	0.69	0.81
50:50	0.63	0.70
25:75	0.65	0.68
0:100	0.86	0.90

4.5 The Variability of Maximum Packing Fraction

A significant parameter proportional to the viscosity of suspensions is the mobility parameter [11]:

$$\phi / (\phi_m - \phi) \quad (4)$$

Here ϕ = Solids volume fraction
 ϕ_m = Maximum packing fraction

ϕ is the total solids volume divided by the total sample volume and ϕ_m is the maximum value of ϕ .

Little correlation occurs between experimental ϕ_m values (Table 4) and viscosity variations. However, experimental ϕ_m values cannot predict solids loadings obtained in this laboratory. ϕ values of 0.45 have been obtained in practice for all the TATB blends listed in the tables although Table 4 indicates that this is impossible for the 100:0, 50:50 and 0:100 blends. ϕ values of 0.48, 0.53 and 0.64 have also been obtained for type A TATB in TNT suspension

although from Table 4 $\phi_m = 0.43$. These higher values of ϕ were obtained by using the technique described in section 3.3. The discrepancies between predicted and observed ϕ seem to be a manifestation of the theory that ϕ_m is shear dependent [12], since TATB incorporation involved shearing the mix. Particle orientation, particle migration leading to optimal packing and aggregate breakup have all been suggested as reasons for the observed shear dependence of ϕ_m [12] but the mechanisms causing the effect in this study are unknown.

Table 4: Maximum Packing Fraction Values for the TATB Blends. Results Determined by Settling Experiments in Molten TNT at 95°C.

Composition of Sample A:B TATB	Maximum Packing Fraction
100:0	0.43
50:50	0.43
25:75	0.47
0:100	0.43

4.6 Comparisons with 60:40 RDX/TNT

RDX/TNT suspensions exhibit considerable long term thickening [5] absent in TATB/TNT. The 60:40 RDX/TNT sample had no yield point. The shear rate drop during 0.1 min at 231 r/min was from 269/s to 204/s and the viscosity increase was from 0.34 Pa.s to 0.43 Pa.s at 231 r/min (c.f. Tables 2 and 3).

The 60:40 RDX/TNT mix possesses the lowest overall viscosity of all the mixes. However, at shear rates less than 50/s (which probably includes the shear rates which occur on pouring), the 25:75 A/B, 50:50 A/B and 75:25 A/B mixes may be brought into approximately the same viscosity range as 60:40 RDX/TNT with little additional energy. This is because most of the thixotropic breakdown takes place at shear rates below 150/s, a practically achievable shear rate.

The 100:0 A/B mix and the 75:25 A/B mix possess the greatest thixotropy and these compositions would require increased power consumption to reduce viscosity. The viscosity of the 0:100 A/B mix cannot be reduced to the same range as 60:40 RDX/TNT at shear rates lower than 50/s after shearing to 231/s and back to 0/s.

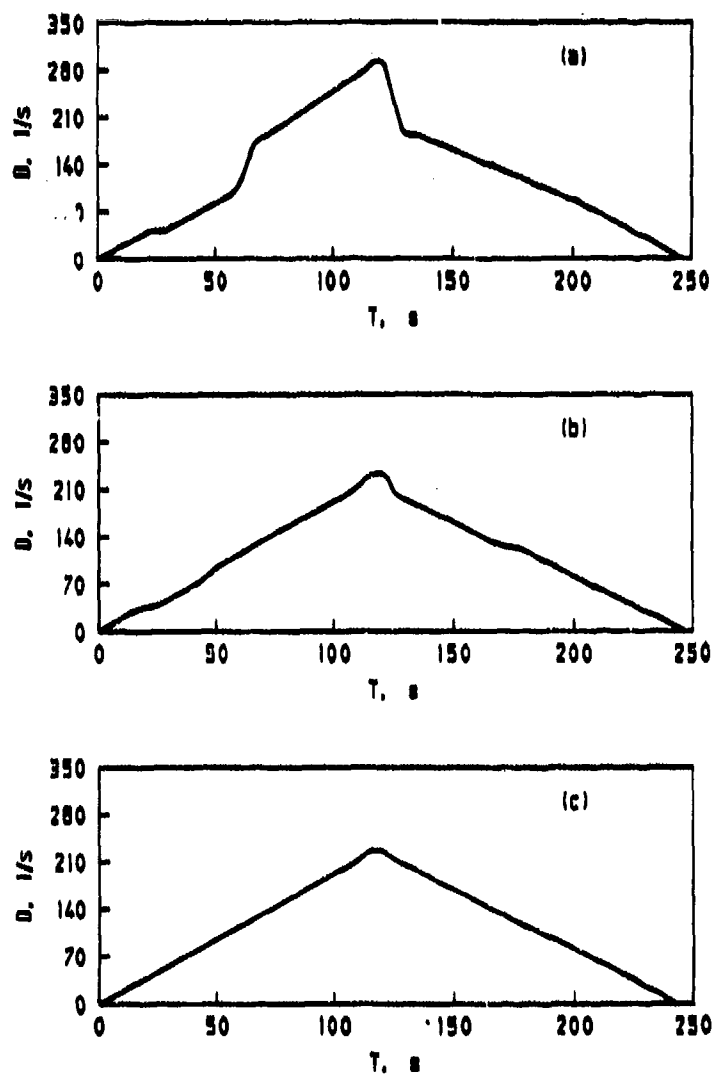


Figure 9: Shear histories for TATB/TNT 51.5/48.5 suspensions at 95°C. (a) TATB type A, (b) TATB A:B = 75.25, (c) TATB A:B = 50:50. Samples sheared from 0 to 231 r/min in 2 min, held at 231 r/min for 0.1 min then back to 0 r/min in 2 min. SVIIP sensor system.

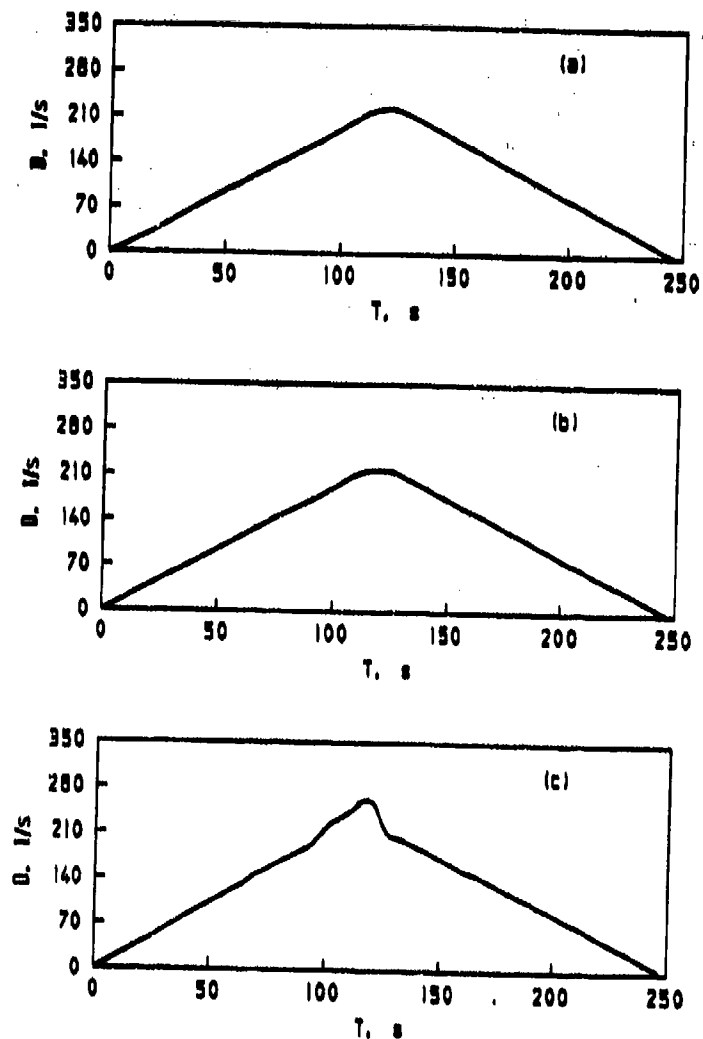


Figure 10: Shear histories for (a) TATB/TNT 51.5/48.5, TATB A:B = 25.75, (b) TATB/TNT 51.5/48.5, TATB type B, (c) RDX/TNT 60/40, RDX grade B suspensions at 95°C. Samples sheared from 0 to 231 r/min in 2 min, held at 231 r/min for 0.1 min then back to 0 r/min in 2 min. SVIIP sensor system.

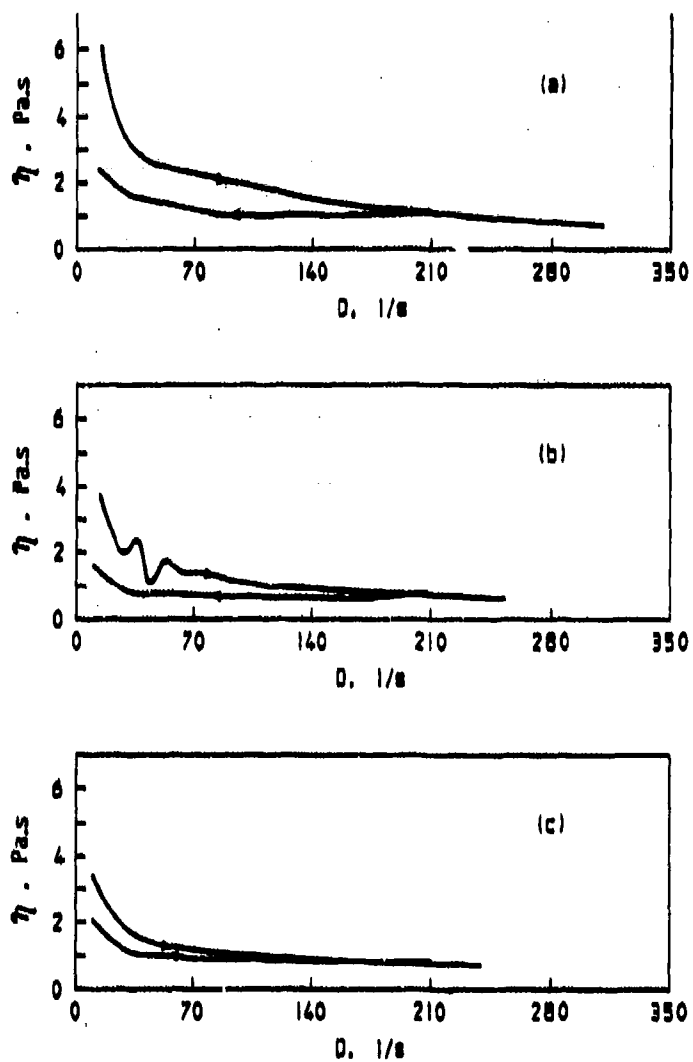


Figure 11: Viscosity vs true shear rate for TATB/TNT 51.5/48.5 suspensions at 95°C. (a) TATB type A, (b) TATB A:B = 75:25, (c) TATB A:B = 50:50. Samples sheared from 0 to 231 r/min in 2 min, held at 231 r/min for 0.1 min then back to 0 r/min in 2 min. SVIIP sensor system.

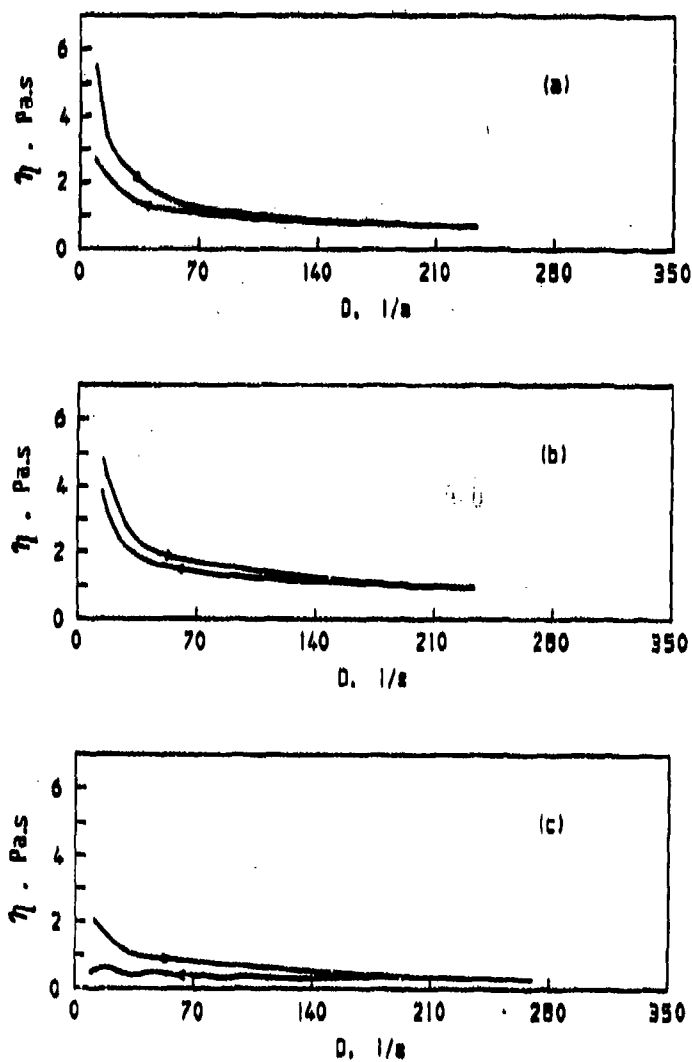


Figure 12: Viscosity vs true shear rate for (a) TATB/TNT 51.5/48.5, TATB A:B = 25/75, (b) TATB/TNT 51.5/48.5, TATB type B, (c) RDX/TNT 60/40, RDX grade B suspensions at 95°C. Samples sheared from 0 to 231 r/min in 2 min, held at 231 r/min for 0.1 min then back to 0 r/min in 2 min. SVIIP sensor system.

5. Conclusions and Recommendations

We conclude that the viscosity of TATB/TNT suspensions may be improved by blending the various types of TATB available. It has been shown that suspensions containing as high as 70 wt percent of TATB type A in molten TNT are possible (this corresponds to the $\phi = 0.64$ composition described in section 4.5). This study shows that the solids loadings and viscosities of such concentrated suspensions can be improved over a range of practical shear rates by appropriately blending type A and type B TATB. The maximum packing fractions of blends of the two types of TATB are shear-dependent and incapable of prediction by standard settling experiments.

It is not possible to give a unique value for the viscosity of a given blend. The reason is that concentrated TATB/TNT suspensions are thixotropic, i.e. their viscosities depend significantly on shear rate and shear application time. Therefore a full characterisation of their flow properties requires thixotropic modelling and this should be conducted to determine processing conditions for optimal viscosity.

The compositions with the greatest apparent thixotropy contained the largest proportions of type A TATB. A further observation is that compositions containing the 25:75, 50:50 and 75:25 type A/type B blends can be brought into approximately the same viscosity range as 60:40 RDX/TNT by application of a shear rate below 150/s.

Performance and vulnerability assessments should be carried out on TATB/TNT castings to provide data on the ability of these materials to meet insensitive munitions requirements.

6. References

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7. List of Symbols Used

Symbol	Meaning
T	Time
ω	Angular velocity of the viscometer bob
τ	Shear stress
η	Viscosity
ϵ	Ratio of the viscometer cup to bob radius
n	Flow behaviour index for a power law fluid
τ_y	Yield stress
D	Shear rate
N	Slope in a plot of $\log \omega$ versus $\log \tau$
ϕ	Solids volume fraction
ϕ_m	Maximum packing fraction, ie the maximum value of ϕ

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The Viscosity of TATB Types A and B Suspensions in Molten TNT: General Characteristics

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ABSTRACT

As part of a program of research into insensitive explosives, an investigation has been conducted into the flow properties of suspensions of TATB types A and B in molten TNT. Low concentration TATB/TNT suspensions are shown to be Newtonian while high concentration suspensions were found to be thixotropic and to exhibit yield point behaviour. The maximum packing fractions of blends of the two types of TATB are shown to be shear-dependent and incapable of prediction by standard settling experiments. The effects on the flow properties of blending the two types of TATB are discussed. Future directions for research are outlined.

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